COLLECTION OF WATER SAMPLES FOR ANALYSIS OF COLLOIDS

Technical Implementing Procedure ID: OSTI-LBNL-TIP/HT-3.0, Rev.0, Mod.0 *Effective: 10/04/04*

1. PURPOSE

This Technical Implementation Procedure (TIP) provides instructions on how to collect and store water samples for subsequent colloid analysis at Lawrence Berkeley National Laboratory (LBNL) for supporting the Office of Science and Technology and International (OSTI)-LBNL Project.

2. SCOPE

This procedure applies to all OSTI-LBNL personnel (or contractor personnel following OSTI-LBNL procedures) involved in the collection and storage of water samples to be used for colloid studies as part of OSTI-LBNL activities subject to the U.S. Department of Energy (DOE) Office of Civilian Radioactive Waste Management (OCRWM) *Quality Assurance Requirements and Description* (QARD), DOE/RW-0333P. Prior to conducting work described in Section 3 of this procedure, personnel performing the sampling work require training in accordance with OSTI-LBNL Quality Implementing Procedure (QIP)-2.0, *Indoctrination and Training of Personnel*.

All technical activities using this procedure shall be in accordance with this TIP. All documentation resulting from actions taken under this TIP shall be recorded in Scientific Notebooks as described in OSTI-LBNL-QIP-SIII.0, *Scientific Notebooks*. While this procedure incorporates specific requirements unique to field sample collection and sample handling, it is consistent with the requirements described in OSTI-LBNL-QIP-SII.0, *Documenting Sample Control*.

If this procedure cannot be implemented as written, OSTI-LBNL personnel shall notify the responsible Principal Investigator (PI) (or designee). If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procedure is modified per OSTI-LBNL-QIP-5.0, *Preparing the Quality Assurance Plan and Quality/Technical Implementing Procedures*.

If the responsible PI (or designee) determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to OSTI-LBNL-QIP-5.0. Such changes are subject to review, usually after the task has proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Scientific staff may use a controlled hard copy or an electronic “Information Only” copy of this procedure (available from the LBNL-ESD website); however, scientific staff are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, controlled copies must be destroyed or marked “superseded” in

accordance with OSTI-LBNL-QIP-6.0, *Controlled Documents*, to ensure that this document is not used to perform work.

3. PROCEDURE

3.1 Principle

This procedure involves collecting water samples from the field for the purpose of subsequent laboratory studies to determine the abundance, composition, size, and morphology of colloids. The collection and sample handling procedure is designed to (i) minimize the potential for contamination of water samples during collection, (ii) obtain sufficient sample for laboratory characterization of colloid samples, (iii) ensure that samples are handled to preserve their colloid properties at the time of collection.

The responsible PI (or designee) decides the sample size to be collected in the field. Typically 2 liters of water are collected for each sample. These amounts shall be documented in the scientific notebook.

In brief, water samples are:

A. Collected from a spring, stream, lake, well, holding tank, water collection system, or other feature of interest;

B. Stored in a completely filled sample bottle;

C. Kept chilled either through the use of a refrigerator or ice chest;

D. Kept out of the light;

E. Transported back to the laboratory as quickly as possible for analysis;

All documentation resulting from actions taken under this TIP shall be recorded in the scientific notebook. A sampling worksheet (Attachment 1) can also be used to record this information; this attachment should be included in the scientific notebook or associated reference binder.

3.2 Equipment

The list of equipment needed to conduct this activity is shown below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the responsible PI (or designee). Any equivalent items used shall be documented in the scientific notebook in accordance with OSTI-LNBL-QIP-SIII.0.

- high-density polyethylene (HDPE) sample bottles
- field collection system (bailer or downhole pump system) for sampling non-artesian wells (not needed for sampling springs and other surface-accessible waters)
- cooler with ice for keeping samples cold

- dark plastic bags for keeping water samples out of light
- 50 ml beakers (for measuring temperature and pH)
- pH paper for making field measurements of pH (including both full range (0-14) and narrow range (e.g., 4-7, 6.5-10) test strips)
- field thermometer (with a range that encompasses 0–100°C) to record sampling temperature of water (either digital thermocouple gauge or pocket dial thermometer with 1°C division).
- tape and indelible marking pen to label sample bottles.

Good field and scientific practices shall be used during sampling to protect against operator injury. If appropriate, personal protection equipment (such as ear plugs, safety glasses, gloves, hard hat, steel-toed boots, sunscreen) may be needed when working at the field site.

3.3 Preparatory Verification

A. Calibration of Measuring and Test Equipment

No measuring and test equipment (M&TE) requiring calibration (per OSTI-LBNL-QIP-12.0, *Control of Measuring and Test Equipment and Calibration Standards*) is needed for this activity. Field measurements of temperature and pH of collected water samples are for reference only, and thus pocket thermometers and pH paper are sufficient for obtaining these measurements.

B. Environmental Conditions

This TIP is performed in the field. Observe and correct factors, if applicable, that may be detrimental to good sample collection, such as swirling dust and rain that could contaminate samples during collection. Record any such conditions and mitigation steps taken in the scientific notebook.

3.4 Control of Samples

It is imperative that sample identification and control be sufficient to trace a sample and its derivatives from its original field location to the point of analysis. For samples collected in the field, a unique identifier is assigned to the sample by the Sample Management Facility (SMF), in accordance with LP-SII.3Q-BSC, *Collection, Submission, Return, and Documentation of Non-Core and Non-Cuttings Samples/Specimens to the Sample Management Facility*. Traceability to this number shall be maintained even if a local identification number is assigned to the sample by recording the field-assigned identifier and the locally assigned identification in the scientific notebook.

3.5 Implementing Procedure

A. Pre-collection Field Activities

It is important to take the appropriate steps to minimize the potential for contamination of colloid samples when collecting samples in the field. There are a number of actions that can be taken to help ensure that collected samples are representative of the features being sampled.

1. When sampling springs, try to obtain samples from the portion of the spring with the highest observed flow. Avoid stagnant portions of the spring if possible. If the spring shows little signs of flow, attempt to sample below the surface while not stirring up sediment from the base of the spring. Note the sampling conditions in the scientific notebook.

2. When sampling water tanks associated with wells, it is best to sample the tank just after it has been filled. Try to sample from the clearest portion of the tank. Note the sampling conditions in the scientific notebook.

3. When sampling non-artesian wells, a bailer or submersible pump is often needed. Make sure that these sampling devices are clean and do not introduce contamination into the well. If possible, try to remove several wetted wellbore volumes of water prior to sampling to minimize the potential for contamination by residual drilling fluids. Record sampling conditions in the scientific notebook.

4. When sampling streams or lakes, try to avoid zones of stagnant water if possible. Do not disturb the bottom when sampling, as this could result in stirring up bottom sediment, which could add colloidal material that is not actually part of the normal suspended material to the collected sample. Record the sampling conditions in the scientific notebook.

B. Sample Collection

Water samples collected from the field for analysis of colloids are normally collected in HDPE bottles and assigned unique sample identifications (e.g., specimen ID, borehole ID, depth interval, collection locality and date).

1. Rinse new HDPE bottles with water from the feature to be sampled. For rinsing, fill the bottle about ¼ full, replace cap, and shake bottle. Pour out water onto lid to rinse inside of lid. Rinse each bottle three times.

2. After rinsing, fill each bottle completely full with water sample, taking care to let all air bubbles escape from the bottle. When all visible bubbles have been removed and no headspace remains in the bottle, screw cap on firmly.

3. Collect a minimum of 2 liters of water for each feature sampled. Be sure to properly label each bottle with unique sample identifications, specifying sample ID, collection locality, date, and in the case of a well, well ID and depth interval. Record sampling information in the scientific notebook.

4. Using 50 ml beakers with water from sampled feature, conduct field measurements of temperature and pH. Note precision of pH paper used to make the measurement (the sensitivity of pH indicator strips is about 0.2 to 0.3 pH units for narrow range (e.g., 4.0–7.0 or 6.5–10) pH paper). Record this information in the scientific notebook.

C. Sample Handling and Storage

The final step of this procedure is to ensure that collected samples preserve their characteristic colloid composition between when they are sampled in the field and analyzed in the laboratory. There are several actions that are required to ensure that minimal sample degradation or modification occurs during transportation back to the laboratory.

1. After the sample has been collected and the bottle labeled, place the bottle inside of a dark plastic bag. Keeping the sample out of the light will retard biological activity that could alter the colloids within the sample.

2. The sample should then be stored within an ice chest. Keeping the sample cool will also retard any biological activity that would potentially affect the colloids within the sample.

3. The sample should then be shipped back to the laboratory as soon as reasonably possible. Preferably, the sample should be sent using an overnight shipping service (it may require several days in the case of international shipments) using an insulated container with ice or some other cooling agent inside.

4. Upon arrival at the laboratory, the sample should be stored in a refrigerator, and analyzed as soon as possible. It is preferable that samples are analyzed within a week of being collected. Note all details regarding the storage, shipping, and handling of samples in the scientific notebook.

3.6 Potential Sources of Error and Uncertainty

There are two main sources of error and uncertainty in the collection and transportation of water samples for colloids. The first source is the possibility that the collected sample is not representative of the feature sample as a result of contamination. As noted in Section 3.5A, the procedure includes steps that reduce the possibility of contamination through rinsing of sample bottles, sampling from the highest flow areas of springs, and pumping out wells prior to sampling to reduce the possibility for contamination.

The second potential source for error and uncertainty is in the degradation of the collected sample between the time of collection and time of analysis. Three steps are taken in the procedure to address this concern. The samples are kept out of the light and cool, and are shipped back to the laboratory as soon as possible.

If a problem occurs during the collection, handling, or transportation of the samples that

could pose a potential source of error or uncertainty for the results, then the staff member shall document it in the scientific notebook and contact the PI about the problem. Samples that have been subjected to nonconforming conditions should be identified as nonconforming and a nonconformance report should be completed as per OSTI-LBNL-QIP-15.0, *Nonconformances*.

3.7 M&TE Storage and Handling

M&TE shall not be handled in a manner that adversely affects its current or future performance. The pH paper should be kept in a Ziploc bag (or in a similar waterproof container) to ensure that it does not get wet prior to its use.

3.8 M&TE Usage

This TIP does not involve any calibration of M&TE (see Section 3.3.A).

3.9 Controls for Out-of-Calibration Conditions

As noted above, the M&TE required for this procedure does not require any calibration. If the temperature or pH of a sample lies outside of the measurement range of the equipment used, then this should be noted in the scientific notebook.

4. RECORDS

The records listed in Section 4.1 shall be collected and submitted the Records Coordinator for submittal to the OCRWM, in accordance with OSTI-LBNL-QIP-17.0, *Records Management*, as individual records or included in a records package.

4.1 Quality Assurance (QA) Records

Scientific Notebooks or associated Reference Binders

4.2 Non-QA Long Term Records

None.

4.3 Non-QA Short-Term Records (three years or less retention)

None.

5. RESPONSIBILITIES

5.1 The **Principal Investigator (PI)** is responsible for assuring full compliance with this procedure and providing training thereof. The PI is responsible for overseeing and coordinating the preparation, review, distribution, revision, and recommending rescission of the TIP.

5.2 Scientific Staff are responsible for following this procedure and turning over related documentation to the Records Coordinator for submittal to the OCRWM, in accordance with OSTI-LBNL-QIP-17.0. Related data shall be turned over to Technical Data Coordinator in accordance with OSTI-LBNL-QIP-SIII.3, *Submittal and Incorporation of Data to the Technical Data Management System*, for entry into the Technical Database Management System (TDMS).

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

DOE	Department of Energy
ESD	Earth Sciences Division
HDPE	High-density polyethylene
HT	Hydrogeological Testing
LBNL	Lawrence Berkeley National Laboratory
M&TE	Measuring and Test Equipment
OCRWM	Office of Civilian Radioactive Waste Management
OSTI	Office of Science & Technology and International
PI	Principal Investigator
QA	Quality Assurance
QARD	Quality Assurance Requirements and Description
QIP	Quality Implementing Procedure
SMF	Sample Management Facility
TDMS	Technical Data Management System
TIP	Technical Implementing Procedure

6.2 Definitions

Colloids: Particles with size ranges between 1 and 450 nm. These particles consist of inorganic materials (such as clays, silica, oxides and hydroxides) and organic matter, and because they have very low settling velocities, they can be transported in water over long distances.

Technical Implementing Procedure: Each TIP describes OSTI-LBNL technical tasks

that (1) are repetitive, (2) are standardized, and (3) can return different results if deviation from the sequence of steps occur.

Wetted Wellbore Volume: The volume of water that corresponds to the volume of the wellbore below the water table. This volume is equal to $\pi r^2 h$, where r is the radius of the perforated liner of the well and h is the thickness of the saturated zone intersected by the well (i.e., the distance between the top of the water table and the bottom of the well).

7. REFERENCES

DOE/RW-0333P, *Quality Assurance Requirements and Description*

LP-SII.3Q-BSC, *Collection, Submission, Return, and Documentation of Non-Core and Non-Cuttings Samples/Specimens to the Sample Management Facility*

OSTI-LBNL-QIP-2.0, *Indoctrination and Training of Personnel*

OSTI-LBNL-QIP-5.0, *Preparing the Quality Assurance Plan and Quality/Technical Implementing Procedures*

OSTI-LBNL-QIP-6.0, *Controlled Documents*

OSTI-LBNL-QIP-12.0, *Control of Measuring and Test Equipment and Calibration Standards*

OSTI-LBNL-QIP-15.0, *Nonconformances*

OSTI-LBNL-QIP-17.0, *Records Management*

OSTI-LBNL-QIP-SII.0, *Documenting Sample Control*

OSTI-LBNL-QIP-SIII.0, *Scientific Notebooks*

OSTI-LBNL-QIP-SIII.3, *Submittal and Incorporation of Data to the Technical Data Management System*

8. ATTACHMENTS

Attachment 1- Data Sheet for “Water Sampling for Colloid Analysis”.

9. REVISION HISTORY

10/04/04 Revision 0, Modification 0
Initial issue.

10. APPROVAL

(Signature on File) _____ _____

Preparer/PI: Patrick Dobson Date

(Signature on File) _____ _____

Technical Reviewer: Rohit Salve Date

(Signature on File) _____ _____

Technical Reviewer: Grace Su Date

(Signature on File) _____ _____

QA Reviewer: Vivi Fissekidou Date

(Signature on File) _____ _____

Project Manager: Gudmundur S. Bodvarsson Date

Water Sampling for Colloid Analysis

Pre-Sampling Steps	
Feature type (e.g., spring, stream, lake, well, tank)	
Feature name	
Feature location	
Feature characteristics (e.g., flow, turbidity)	
If well, estimate wetted wellbore volume	
If well, estimate amount of fluid lifted from well prior to sampling	
Feature comments	
Sampling Steps	
Sampling method	
Bottle prerinse	
Time of sampling	
Amount collected	
Sample description	
Sample ID	
Measured T (°C)	
Measured pH	
Sampling comments	
Handling Steps	
Field storage details	
Shipping details	
Date sample arrived in lab	
Date sample analyzed	
Handling comments	